IS 3241 (1985): Geranyl Acetate [PCD 18: Natural and Synthetic Fragrance Materials]

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Indian Standard

SPECIFICATION FOR GERANYL ACETATE

(First Revision)

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Indian Standard

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(First Revision)

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Indian Standard

SPECIFICATION FOR GERANYL ACETATE

(First Revision)

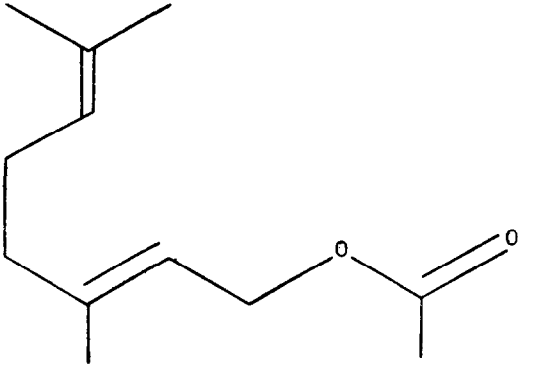
0. FOREWORD

0.1 This Indian Standard (First Revision) was adopted by the Indian Standards Institution on 25 March 1985, after the draft finalized by Natural and Synthetic Perfumery Materials Sectional Committee had been approved by the Petroleum, Coal and Related Products Division Council.

0.2 This standard was first published in 1965. The Sectional Committee responsible for its preparation felt that the standard should be revised with a view to bringing it in line with trade practices in perfumery technology and also to align the quality level of the material currently manufactured and sold in the country which is given here for guidance only.

0.3 In this revision, the gas chromatographic method which is being progressively used in the country has been included and the wet method of analysis for determination of ester content has been deleted.

0.4 Geranyl acetate ($C_{12}H_{20}O_2$) is the acetate of primary allyl alcohol, geraniol. It is also known as 2-(trans)-3, 7-dimethyl-2, 6 octadiene-1-yl-acetate. It is represented by the following structural formula:



GERANYL ACETATE
(Molecular Mass 196·28)

0.5 Geranyl acetate is distributed widely in nature and occurs in oils of *Daucus carota, Eucalyptus macarthurii, Eucalyptus staigeriana* and to a smaller extent in oils of geranium, citronella (Java and Formosa types), palmarosa, neroli, petitgrain and others. However geranyl acetate produced commercially in India is obtained from geraniolex citronella oil which itself is a natural mixture of geraniol and citronellol. Hence, geranyl acetate available at present in India is mainly a mixture of geranyl acetate and citronellyl acetate.

0.6 The material is widely used in different perfumery compounds and mainly in conjunction with geraniol, the parent alcohol in rose compositions and for artificial oils of geranium and lavender. It is extensively used for compounding many kinds of perfumes for cosmetics, toiletries and soaps, and in the blending of artificial essential oils and flavours.

0.7 In the preparation of this standard, considerable assistance has been derived from the following publications:

EOA No. 11-1975 Standard for Geranyl Acetate. Essential Oil Association of USA, New York.

The Givaudan Index, 1978. Givaudan-Delawanna, Inc., New York.

0.8 For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS : 2-1960*. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

1. SCOPE

1.1 This standard prescribes the requirements and the methods of sampling and test for the material commercially known as geranyl acetate obtained from oil of citronella (ex citronella oil).

2. TERMINOLOGY

2.1 For the purpose of this standard, definitions given in IS : 6597-1972† shall apply.

3. REQUIREMENTS

3.1 Description

3.1.1 The material is exclusively manufactured by acetylation of geraniol ex citronella oil (*see* Type 2 of IS : 1800-1961‡). The acetylated product shall have been carefully fractionated.

*Rules for rounding off numerical values (*revised*).
†Glossary of terms relating to natural and synthetic perfumery materials.
‡Specification for geraniol.

3.1.2 The material shall be a clear and colourless liquid, free from sediment, suspended matter and adulterants.

3.1.3 The material shall also be tested olfactorily and especially for by-notes and for the presence of adulterants and impurities, if any, as prescribed under **4** and **5** of IS : 2284-1963*.

3.2 Solubility — The material shall be soluble in 8 volumes of ethanol (70 percent by volume), when tested as prescribed under **8** of IS : 326-1968†.

3.3 The material shall also comply with the requirements given in Table 1.

4. PACKING AND MARKING

4.1 Packing — The material shall be supplied in amber-coloured glass or other air-tight opaque tin-lined or aluminium containers. Suitable galvanized iron containers may also be used.

4.1.1 The material shall be protected from light and stored in a cool and dry place.

4.2 Marking — The material shall be marked with the following information:

a) Name of the material;
b) Name of the manufacturer and recognized trade-mark, if any;
c) Net mass of the material; and
d) Batch number.

4.2.1 The containers may also be marked with the ISI Certification Mark.

NOTE — The use of the ISI Certification Mark is governed by the provisions of the Indian Standards Institution (Certification Marks) Act and the Rules and Regulations made thereunder. The ISI Mark on products covered by an Indian Standard conveys the assurance that they have been produced to comply with the requirements of that standard under a well-defined system of inspection, testing and quality control which is devised and supervised by ISI and operated by the producer. ISI marked products are also continuously checked by ISI for conformity to that standard as a further safeguard. Details of conditions, under which a licence for the use of the ISI Certification Mark may be granted to manufacturers or processors, may be obtained from the Indian Standards Institution.

*Method for olfactory assessment of natural and synthetic perfumery materials.
†Methods of sampling and test for natural and synthetic perfumery materials (*first revision*).

TABLE 1 REQUIREMENTS FOR GERANYL ACETATE

(*Clauses* 3.3 *and* 6.1)

Sl No.	Characteristic	Requirement	Methods of Test, Ref to	
			Indian Standard	Appendix
(1)	(2)	(3)	(4)	(5)
i)	Odour	Fresh, dry rosepetal like note	IS : 2284-1963*	
ii)	Relative density at 27/27°C (*see* Note 1)	0·899 2 to 0·904 9	IS : 326 (Part 3)-1980†	
iii)	Optical rotation	± 1°	IS : 326 (Part 4)-1980†	
iv)	Refractive index at 27°C (*see* Note 2)	1·452 to 1·458	Cl 7 of IS : 326-1968‡	
v)	Acid value, *Max*	1·0	IS : 326 (Part 7)-1980†	
vi)	Gas chromatographic analysis:		—	A
	a) Geranyl acetate, percent by mass, *Min*	69		
	b) Citronellyl acetate, percent by mass, *Max*	25		

NOTE 1 — The correction factor for relative density for each degree celsius change in temperature is 0·000 64.

NOTE 2 — The correction factor for refractive index for each degree celsius change in temperature is 0·000 38.

*Method for olfactory assessment of natural and synthetic perfumery materials.

†Methods of sampling and test for natural and synthetic perfumery materials:
Part 3 Relative density (*second revision*).
Part 4 Determination of optical rotation (*second revision*).
Part 7 Determination of acid value.

‡Methods of sampling and test for natural and synthetic perfumery materials (*first revision*).

5. SAMPLING

5.1 Representative samples of the material, each sample containing not less than 50 ml shall be drawn as prescribed in IS : 326 (Part 1) - 1984*.

6. TEST METHODS

6.1 Tests shall be carried out as prescribed under **3.1**, **3.2**, **3.3** and the appropriate references specified in col 4 and 5 of Table 1.

*Methods of sampling and test for natural and synthetic perfumery materials: Part 1 Sampling (*second revision*).

6.2 Quality of Reagents — Unless specified otherwise, pure chemicals and distilled water (*see* IS : 1070-1977*) shall be employed in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

APPENDIX A

[*Table* 1, *Item* (vi)]

GAS CHROMATOGRAPHIC ANALYSIS OF GERANYL ACETATE

A-0. GENERAL

A-0.1 The chromatographic conditions given here are for guidance only.

A-0.2 Outline of the Method — A sample of the material is injected into the gas chromatograph where it is carried by the carrier gas from one end of the column to the other. During its movement the constituents of the sample undergo distribution at different rates and ultimately get separated from one another. The separated constituents emerge from the end of the column one after another and are detected by suitable means whose response is related to the amount of a specific component leaving the column.

A-1. APPARATUS

A-1.1 Any gas chromatograph capable of being operated under conditions suitable for resolving the individual constituents into distinct peaks may be used. A typical chromatograph for Geranyl acetate using a chromatograph with the following chromatographic conditions is shown in Fig. 1.

Sample	Geranyl acetate
Column	
a) Material	Stainless steel
b) Length	2·438 m
c) OD	7·95 mm
d) ID	6·35 mm
e) Stationary phase	Carbowax 20 M, 10 percent by mass
f) Solid support	Chromosorb WAW 80-100 mesh

*Method for olfactory assessment of natural and synthetic perfumery materials.

Carrier Gas	Nitrogen
Conditions	
a) Column temperature iso-thermal	175°C
b) Injection port temperature	250°C
c) Carrier gas flow rate	26 ml/min
d) Inlet pressure	3 kg/cm^2
Detector	
a) Type	Flame ionisation
b) Temperature	250°C
Recorder	
a) Span	21·8 cm
b) Chart speed	0·5 cm/min
Attenuation	15

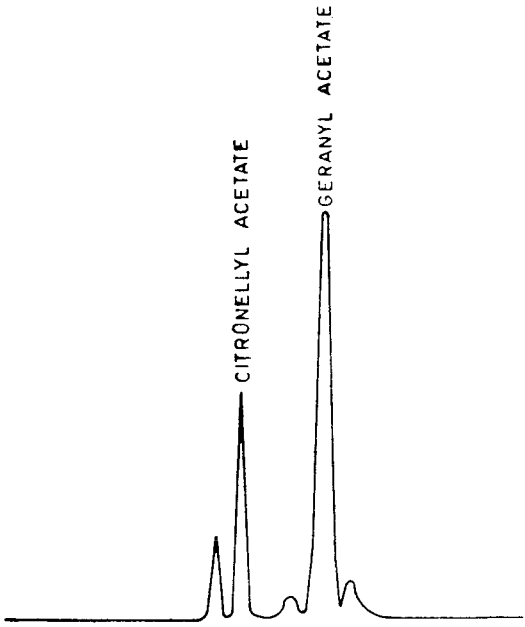


FIG. 1 TYPICAL CHROMATOGRAM OF GERANYL ACETATE

A-2. PROCEDURE

A-2.1 Conduct the flow of the carrier gas and inject the sample at inject port where it is vaporized and well mixed with the carrier gas. This is led into the chromatographic column wherein vaporized constituents of the sample are separated out by virtue of their differing interaction with the stationary phase. As the different constituents pass through the detector, they give signals corresponding to the amount of particular constituents leaving the column. The detector signal, on transmission to the recorder, plots the chart. From the specific area under various peaks corresponding to specific constituents, the quantities of different constituents are determined.

NOTE — For the separation to be efficient, it is necessary that the column is maintained at the temperature suggested throughout the time required for the resolution of the constituents.

A-3. CALCULATION

A-3.1 Area Measurement (*see* **Note 1**) — Since normal peaks approximate a triangle the area is measured by multiplying the peak height times the width of half height. The normal peak base is not taken since large deviations may be observed due to tailing or adsorption. This technique is rapid, simple and fairly accurate when peaks are symmetrical and of reasonable width.

A-3.2 Area Normalization (*see* **Note 2**) — By normalizing, it meant, calculating the percentage composition by measuring the area of each and dividing the individual areas by total area, for example,

$$\text{Percentage of } A = \frac{\text{Area of } A}{\text{Total area}} \times 100$$

NOTE 1 — Other methods of area measurements, namely, Triangulation, Disc Integrator and Electronic Digital Integrator if fixed with GLC machine would be of great advantage.

NOTE 2 — Internal standardization can be used if pure appropriate internal standard is available. This method is known as relative or indirect calibration.

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